

# PATENT SPECIFICATION

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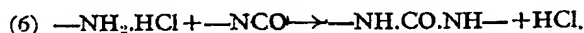
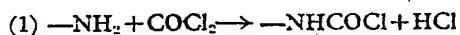
## (54) PRODUCTION OF ISOCTANATES

(71) We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, of Imperial Chemical House, Millbank, London, S.W.1., a British Company, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to the manufacture of organic mono- and polyisocyanates from organic amines and polyamines by a phosgenation process.

The manufacture of organic isocyanates by phosgenation of amines and polyamines is well-known and although it may be carried out either batchwise or continuously, either process involves an initial stage at which the amine or polyamine and phosgene are brought into contact one with another. In this initial or first stage many reactions may occur concurrently and the final yield of isocyanate or polyisocyanate is considerably influenced by the reactions which occur in the first stage.

The following are typical of the reactions of the amine group which may take place during the initial mixing stage.

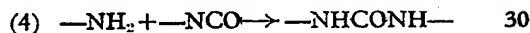
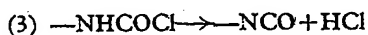


Reactions (4) and (6) are most undesirable as substituted urea groups once formed react only very slowly and incompletely with phosgene to yield isocyanates as follows:



Substituted ureas also react with phosgene and hydrogen chloride to form small but significant amounts of products containing chlorine. These impurities may persist in the final isocyanate product and be responsible for high acidity and a high content of hydrolysable chlorine, both of which may adversely

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Reaction (1) the formation of carbamyl chloride and reaction (3) the decomposition of carbamyl chloride to give isocyanate are both rapid reactions and do in fact take place sufficiently rapidly for reactants (2) and (4) which make use of the products of reactions (1) and (3) to take place almost immediately on mixing the polyamine and phosgene.

Reaction (2) is to some extent undesirable, as although the amine hydrochloride group can be converted to the isocyanate group by hot phosgenation, it is essential that the amine hydrochloride be in a fine physical form or phosgenation is unduly prolonged, this is particularly important in the case of a continuous process.

Not only does coarse amine hydrochloride phosgenate slowly at the hot stage of the process but it can take part at high temperatures in a further undesirable reaction involving formation of urea groups e.g.

influence the technical applications of the isocyanate product.

The formation of urea groups is therefore undesirable from the point of view both of yield and quality of the desired product.

Thus, in order to obtain optimum conditions for the formation of polyisocyanate in

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good yield it is essential that the following conditions be satisfied in the initial mixing stage of phosgenation.

5 a) A high concentration of phosgene must be present in the reaction zone in order to maximise reaction (1) and minimise reaction (4).

10 b) Any amine hydrochloride produced as a result of reaction (2) must be in a satisfactory physical form to enable it to react readily with phosgene at any subsequent hot stage.

15 It has been found that the freshly prepared amine hydrochloride rapidly becomes more coarse and delay between its initial formation at the cold stage and its subsequent phosgenation at the hot stage must be minimised.

20 c) Contact of free amine with free isocyanate groups must be avoided as far as possible in order to prevent formation of urea groups by reaction (4).

25 It has now been found that these conditions can be met in the initial reaction if phosgene is reacted with a solution of an amine or a polyamine in an inert solvent and the solution of amine or polyamine is in the form of a forward flowing agitated film ensuring little or no back-mixing. The product from this first stage consists essentially of a mixture of isocyanate, amine hydrochloride, carbamyl chloride, phosgene, hydrogen chloride and solvent and it is a further essential feature of the present process that this mixture be discharged directly into a hot reactor.

40 It has been found that by using the amine or polyamine solution in the form of a forward flowing agitated film, reaction may take place both on the surface and also in solution due to dissolution of phosgene and that agitation of this film, whose surface area/volume ratio is much greater than that in a normal reaction vessel not only aids rapid dissolution of phosgene but also permits easy escape of hydrogen chloride and in regard to amine hydrochloride formed by reaction (2) gives the hydrochloride in a very fine physical form eminently suitable for phosgenation. Reaction in a forward flowing film tends to carry any isocyanate formed away from the fresh amine and thus prevents reaction of these two according to reaction (4) to give undesirable ureas.

55 It has also been found possible using the present process to employ higher concentrations of polyamine than have hitherto been found useful and the process has also been found adaptable for polyamines which by previous methods gave practically unstirrable slurries on first stage treatment with phosgene.

60 Thus according to the present invention there is provided a process for the continuous manufacture of organic isocyanates by the

65 phosgenation of organic amines or polyamines wherein the first stage comprises reacting an excess of phosgene with an amine or polyamine in solution in an inert solvent, the amine or polyamine being in the form of a forward flowing agitated film of a solution of the amine or polyamine in an inert solvent, the product of this first stage being discharged directly into a hot reactor.

70 The forward flowing agitated film of amine or polyamine solution may be produced by a variety of methods, thus, the solution may be allowed to flow down an inclined surface or by pressure of liquid behind it may be made to flow along a horizontal surface. The film will be agitated to some degrees by the mere movement of flow but it may be further agitated mechanically or by creating turbulence in the atmosphere above the surface.

80 The inner surface of a cylindrical tube has been found to be a particularly suitable surface on which to form the film of amine solution, the tube may be horizontal or may be set at an angle to horizontal, an angle of less than 30 degrees being preferred.

90 The amine or polyamine solution may be fed into one end of the cylindrical tube and the desired film maintained flowing along the inner wall of the tube by agitation within the tube, thus the solution can be fed onto the wall of the tube and may be progressed along the tube in a helical path by blades, arms or sweeps mounted on a rapidly rotating shaft aligned centrally down the tube. The blades, arms or sweeps may be so adjusted to pass through the film of liquid, this is preferred, or they may be so adjusted to merely agitate the surface by passing close thereto. In order to obtain any amine hydrochloride formed in a very fine state of subdivision it is preferable to agitate within the liquid film to comminute the particles thereof. The force exerted by a rotating assembly of this nature automatically returns to the film any particles of solution or slurry which leave the film. The solution on reaction with the phosgene changes into a slurry of fine particles and at the end of this first stage is discharged directly into a hot reactor.

115 The cylindrical tube may be jacketed to permit cooling or heating during the phosgene/amine or polyamine reaction, alternatively the reaction which is exothermic may be allowed to proceed adiabatically.

120 In addition to at least one inlet for amine and inert solvent, at least one inlet is provided for feeding phosgene, the phosgene may be in the form of gas, may be liquefied or may be in solution in inert solvent. The tube may be also be provided with an outlet for refluxing phosgene boiled off in the reaction and returning it directly to the reactor.

125 In order to assist in direct discharge of the product of the first stage into a hot reac-

tor the cylindrical tube is preferably open ended at the end away from the inlets.

When the cylindrical tube is sloping the amine solution flows down the inner surface of the tube under the force of gravity, when it is in a horizontal position the feed of amine may be pumped in and the feed pressure utilised to push the film along the tube.

Tubes with tapering sides, for example in the form of a conic frustum may also be used.

One type of apparatus available commercially which may be adapted for carrying out the first stage of the present process is that generally known as a thin film processor, although in the present case it is not essential that the film of amine or polyamine solution be a thin film.

Processors of this type which may be used for the first stage of the present process include the reverse-taper horizontal thin-film processor manufactured by the Kontro Company Inc., of Petersham, Massachusetts, U.S.A.

Reaction between phosgene and amine or polyamine in the first stage may take place at any temperature but is conveniently carried out between 8°C and 100°C

The first stage may be carried out under atmospheric pressure or under pressures higher than atmospheric and in order to increase solubility of phosgene in the solvent it is preferred to operate at pressures of from 2.5 lbs./sq. in. to 250 lbs./sq. in. The remainder of the phosgenation may be operated at atmospheric or higher pressures but it will be obvious that the hot reactor into which the product of the first stage is discharged direct must operate at the same pressure as the vessel in which the first stage is carried out.

The residence time in the first stage vessel may be varied but is preferably adjusted to obtain the highest throughput conditional on all the amine feed having been dispersed and reacted before the product is discharged into the hot reactor. Appropriate residence times range between 2 and 10 minutes.

Direct discharge of the product of the first stage into the hot reactor may be achieved by having the cylindrical tube opening directly into the side of the reactor or by having it connected thereto by a wide vertical tube down which the slurry may pass into the hot reactor, assisted if necessary due to the thickness of the slurry by a screw conveying device. It is an essential feature of the invention that passage should be by direct connection and that the slurry should not be passed by ordinary pipework connections to the hot reactor.

The hot reactor should be of such a capacity that as the product of the first stage is added thereto, it is immediately heated to the temperature of the hot reactor.

The hot reactor may be any normal type of heated phosgenation vessel such as are well

known in the art for continuous or semi-continuous phosgenation processes. The hot reactor may be operated at any temperature in excess of about 80°C. and may be the reactor in which phosgenation is completed or may be the first of a train of reactors operated at temperatures above 80°C.

Thus after the first stage, the phosgenation may be completed by any of the methods known in the art for continuous phosgenation processes, the size and number of the reactors used being determined solely by the throughput desired and the residence time required.

Further phosgene may be added in the hot reactor or at any later stage as desired. Phosgene covered from the off-gases may be used at any stage.

On completion of the phosgenation the final product may be isolated in the crude or pure state by known methods.

The drawings accompanying the Provisional Specification illustrate apparatus found suitable for carrying out the process of the present invention.

The invention will now be described with particular reference to the drawings in which Figures 1 and 2 relate to apparatus suitable for carrying out the present process on the small scale and Figures 3 and 4 relate to vessels suitable for operation on a large scale.

Figure 1 is an elevation of a cold (first) stage reactor with hot stage reactor attached.

Figure 2 is a perspective view and end elevation of an agitator for the cold stage reactor shown in Figure 1.

Figures 3 and 4 are pictorial representations of reactors suitable for carrying out the present process on the large scale.

In Figure 1 the cold or first stage reactor comprises the arm Q of the cruciform shaped vessel, which arm Q is directly connected to a hot reactor I by the lower arm R of the cruciform vessel. The arm Q carries two inlets A and B adapted for the admission of amine solution and phosgene respectively. Provision (not shown in the drawing) may also be made in the upper surface of the arm Q for a further inlet/outlet connected to a reflux condenser to permit reflux of phosgene from the initial reactor, arm Q. The vertical axes of the arms Q and R are conveniently slightly offset as shown in the cross section at the line XX, to facilitate the fitting of agitators in each arm.

An agitator of the type shown in Figure 2 is carried with its axis along the horizontal axis of the arm Q, the blades of the agitator being of such a length that they extend from the point K to the point J. The agitator as shown in Figure 2 comprises a stainless steel shaft, preferably of a type known to resist corrosion under phosgenation conditions, to which two spiders G are attached, the two spiders carrying between them 4 hinged stainless steel blades H. The blades H are so

mounted that when the shaft is rotated the four blades swing on rotation to wipe the wall of the initial reactor Q promoting good mixing in the film of liquid thereon and turbulence in the central area of Q.

A further agitator may be mounted vertically in the arm R and may be of the type illustrated in Figure 2; alternatively a scroll may be mounted vertically in the arm R. The purpose of the agitator or scroll is to assist in conveying the product of the initial reaction in Q directly to the hot reactor I. The initial product leaving Q is very thick and this second agitator or scroll prevents blockages in the direct feed.

When the apparatus depicted in Figure 1 is in use, phosgene and amine solution are fed in at the respective inlets with the agitator in the arm Q rotating at about 250 to 950 r.p.m. and the vertical agitator in the arm R rotating at about 100 to 250 r.p.m. Reaction between the phosgene and the amine takes place as the amine solution flows along the inner surface of the arm Q towards the end J, the rate of flow being so adjusted that the initial or first reaction stage has been completed by the time the solution, or suspension as it then is, passes directly from the initial reactor into the hot reactor via the arm R. Phosgenation is then completed in the hot reactor and product continuously withdrawn via a dip-leg (not shown) through one of the ports C, D or F. If desired the product from the hot reactor can be passed to one or more further hot reactors for completion of phosgenation in a series of stages.

Referring now to Figures 3 and 4, the initial stage reactor is comprised of the tapered vessel L fitted with an agitator comprised of blades or sweeps M driven by the motor N. Inlets for phosgene P and amine solution S are provided in the upper surface of the tapered vessel L. The tapered vessel may be jacketed and a further port may be provided in the upper surface of L to permit reflux of phosgene if desired.

In Figure 3 the tapered vessel L is mounted on a hot phosgenator O and the exit from the tapered vessel opens directly into a top flange on the hot phosgenator. In the variation shown in Figure 4 the tapered vessel is mounted on the side of a hot phosgenator and the exit opens directly into the hot phosgenator.

A multiplicity of inlets for phosgene and amine solution into the tapered vessel are preferably provided and although not essential it is preferred that the phosgene inlets are the furthest away from the exit to the hot phosgenator.

The vessels L and M and ancillary apparatus are constructed of materials known to be resistant to corrosion under phosgenation conditions.

The hot phosgenator M is provided with a bottom run off T or alternatively with a side

outlet (not shown). Material withdrawn from the hot phosgenator may be worked up and the product isolated in the normal manner if phosgenation is complete or may be passed continuously to a further phosgenator as desired.

The large scale equipment illustrated in Figures 3 and 4 is operated in a similar manner to the small scale apparatus already described.

The present process is for continuous operation but may be operated semi-continuously, that is the first stage is operated continuously until a later hot reactor is fully charged, the first stage operation is then suspended and the material in the later hot reactor phosgenated batchwise to completion, the product discharged and the whole operation restarted.

Organic isocyanates and polyisocyanates which may be manufactured by the present process include aromatic, aliphatic, cycloaliphatic and araliphatic mono- and polyisocyanates and include for example hexyl, cyclohexyl, benzyl, phenyl, chlorophenyl and naphthyl isocyanates, tolylene diisocyanates, in particular mixtures of the 2,4- and 2,6- isomers, diphenylmethane - 4,4' - diisocyanate and mixtures thereof containing polyisocyanates of higher functionality, 3 - methyl-diphenylmethane - 4,4' - diisocyanate, meta and para phenylene diisocyanates, diphenyl - 4,4' - diisocyanate, naphthalene - 1,5 - diisocyanate, chlorophenylene - 2,4 - diisocyanate, tetramethylene diisocyanate, pentamethylene diisocyanate, hexamethylene diisocyanate, heptamethylene diisocyanate, octamethylene diisocyanate, nonamethylene diisocyanate, decamethylene diisocyanate, dodecamethylene diisocyanate, m - xylene diisocyanate, p - xylylene diisocyanate, cyclohexane - 1,4 - diisocyanate, cyclohexane - 1,3 - diisocyanate, methylcyclohexane - 2,4 - and 2,6 - diisocyanates and mixtures thereof, 1,3 - and 1,4 - bis (isocyanatomethyl)cyclohexane, diisocyanatodicyclohexylmethane,  $\beta$  - isocyanato - ethylphenyl isocyanate,  $\alpha$  - isocyanato - benzylisocyanate and tetramethyl - p - xylylene diisocyanate.

The above listed isocyanates are manufactured using corresponding well known primary amines as starting materials.

The present process has been found to be particularly applicable to the manufacture of organic polyisocyanates both aromatic and aliphatic in character, giving unexpectedly high yields and short phosgenation times.

The inert solvent employed in the present process may be any of those solvents such as have heretofore been proposed for use in phosgenation reactions. The term inert means inert under the reaction conditions, thus the solvent must obviously be inert to hydrogen chloride, phosgene and the amine under the reaction conditions employed.

Inert solvents include ester solvents such

as n - butyl acetate, n - hexyl acetate, dimethyl carbonate and diethyl carbonate, hydrocarbons such as toluene and xylene, and halogenated hydrocarbons such as mono-, di- and trichlorobenzenes chlorotoluenes, carbon tetrachloride and trichlorofluoromethane.

The strength of the solution of the amine in the inert solvent, hereinafter referred to as the amine solution may vary widely but it is preferred to use a solution containing between 4% and 16% by weight of the amine. In the present process it has been found that amine and polyamine solutions containing up to 10% by weight of the amine and polyamine can conveniently be used.

An excess of phosgene is always present in the first stage and by an excess is intended an excess over that required stoichiometrically for reaction with all the amine groups present at that particular time.

The invention is illustrated but not limited by the following examples in which all parts and percentages are by weight.

#### EXAMPLE 1

##### Preparation of polyamine

465 Parts aniline, 300 parts water and 499.5 parts hydrochloric acid (36%) are mixed together with good agitation. The temperature rises from approximately 20°C to 60°C. To the clear solution is added 242.4 parts formaldehyde (36.5%) during 30 minutes, the temperature rising to approximately 90°C. The suspension obtained is heated to 95 ± 2°C in 1 hour and held at this temperature for 3 hours. The clear solution is cooled to 60°C and neutralised. After agitation for 30 minutes the upper aqueous layer is run to waste and the bottom layer of polyamine is washed with three successive quantities of 350 parts water at 60°C. The washed product is heated to 115°C under reduced pressure (20 mm Hg) to dry it. The yield is approximately 460 parts.

##### Phosgenation

400 Parts polyamine is dissolved in 600 parts monochlorobenzene at 60°C. 4000 Parts monochlorobenzene at 30°C is added to the solution and after thorough mixing, the solution is filtered and pumped continuously at the rate of 13 g./min. to inlet A of the horizontal arm of the cold stage reactor as shown in Figure 1. The horizontal arm of the reactor is fitted with efficient agitation, an agitator of the type shown in Figure 2 being used. It is operated at 800 r.p.m. Liquid phosgene is fed initially at the rate of 4.5 g./min. to inlet B, when steady running conditions are established, the feed of fresh phosgene is reduced to 3.5 g./min. The temperature of the reactor is 30—35°C.

The thick suspension of reaction mixture flows down the vertical arm of the cold stage reactor, which is equipped with a similar type

of agitator to the horizontal arm but operated at 200 r.p.m., into a vessel of 1000 cc. capacity and approximately 750 cc. working volume. This vessel is fitted with moderate agitation (200 r.p.m.) and is maintained at 85°C. The contents of this reactor overflow to a hold-vessel which is not shown in the diagram.

The off-gases from the hot stage reactor pass through a water-cooled reflux condenser which returns solvent to the hot-stage reactor and then to the base of a condenser operating at -50°C. This condenser separates HCl gas from phosgene which is liquefied. The latter flows, together with the make-up of fresh phosgene feed back to the inlet B in the horizontal arm of the cold-stage reactor.

The dilute crude product which flows from the hot-stage reactor is clear and free from sediment. It is worked up by conventional means, the solvent being evaporated under reduced pressure at temperatures not exceeding 140°C.

The yield is 500 g. of a clear pale brown liquid which contains diphenylmethane - 4,4'- and 2,4' - diisocyanate together with higher homologous polyphenylene methylene - bridged polyisocyanates. The product is suitable for making polyurethane rigid foams, elastomers, sealants, plastics, surface coatings, adhesives and for many other purposes.

#### EXAMPLE 2

A 8% wt./wt. solution of tolylene diamine in o - dichlorobenzene at 70°C is fed into inlet B of the horizontal arm of the cold stage reactor shown in Figure 1. The diamine is a mixture of isomers comprising approximately 80% 2,4-, 19.6% 2,6- and 0.4% 3,4 - tolylene diamine. The reactor is operated in a similar manner to the procedure described in Example 1. The initial rate of feed of fresh phosgene is 4.5 g./min. but when steady running conditions are established and a steady return of phosgene from the hot stage reactor is obtained, the rate of fresh feed of phosgene is reduced to 3.5 g./min. The temperature of the reactor is 25—30°C.

The cold stage reaction mixture flows to the hot stage reactor down the vertical arm, any blocking being prevented by the action of the vertical agitator. From the hot stage reactor, which has a working volume of 750 cc. the dilute crude tolylene diisocyanate flows to a receiving vessel, not shown in the diagram. After filtration to remove a small amount of insoluble residue, the crude isocyanate is degassed to eliminate phosgene and hydrogen chloride. The solvent is removed under reduced pressure in conventional manner and the isocyanate distilled to give a colourless product containing approximately 80% tolylene - 2,4 - diisocyanate and 20% tolylene - 2,6 - diisocyanate.

The yield is 93% and the product is suit-

able in every way for making polyurethane flexible foams, surface coatings and other products.

### EXAMPLE 3

#### 5 Preparation of polyamine

1116 Parts aniline, 160 parts water and 367 parts hydrochloric acid (36.5%) are mixed together with good agitation. The temperature rises from approximately 20°C. to 53°C. To the aqueous mixture of aniline and aniline hydrochloride is added 556 parts formaldehyde (36.0%) during 30 minutes. The temperature rises during the addition and is controlled at 75–80°C. by the application of external cooling. After addition of formaldehyde is complete, the suspension obtained is heated to  $95 \pm 2^\circ\text{C}$ . in 15 minutes and held at this temperature for 90 minutes. The clear solution is neutralised by adding a solution of 150 parts caustic soda in 300 parts water. After agitation for 30 minutes the upper aqueous layer is run to waste and the bottom layer of polyamine is washed with three successive quantities of 800 parts water at 80°C.

The washed product is treated at 115°C. under reduced pressure (20 mms. Hg) in a falling film evaporator to remove water and excess aniline.

30 The yield is approximately 1025 parts.

#### Phosgenation

960 Parts of the above polyamine is dissolved in 7040 parts monochlorobenzene at 60°C. The clear solution is cooled to 30°C. and phosgenated continuously in the manner described in Example 1.

The dilute product is de-gassed and the product worked up by conventional means, the solvent being removed under reduced pressure.

40 The concentrated material is heated to 195°C. held at this temperature for 15 minutes, and cooled rapidly to below 100°C.

45 The product is a clear brown liquid and has the following analytical characteristics:  
Isocyanate value (as MDI): 89.2% (calculated to MW 250)

4,4' - Diphenylmethanediisocyanate content:	42.3%
50 2,4' - Diphenylmethanediisocyanate content:	2.3%
Hydrolysable chlorine content:	0.37%
Acidity (as HCl)	0.086%
55 Soluble dimer content:	1.1%
Viscosity:	281 C/S

The product is suitable for making rigid, flexible and semi-flexible foams, elastomers, sealants, plastics, surface coatings, adhesives and for many other purposes.

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#### WHAT WE CLAIM IS:—

1. A process for the continuous manufacture of organic isocyanates by a two stage phosgenation of amines or polyamines wherein the first stage comprises reacting an excess of phosgene with an amine or polyamine in solution in an inert solvent, the amine or polyamine being in the form of a forward flowing agitated film of a solution of the amine or polyamine in an inert solvent, the product of this first stage being discharged directly into a hot reactor.

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2. A process as claimed in Claim 1 wherein the forward flowing agitated film of a solution of the amine polyamine in an inert solvent is formed on the inner surface of a cylindrical tube.

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3. A process as claimed in Claim 2 wherein the amine or polyamine solution is fed into one end of a cylindrical tube and the desired film maintained flowing along the inner wall of the tube by agitation means provided within the tube.

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4. A process as claimed in Claim 3 wherein the agitation means comprise blades, arms or sweeps mounted on a rapidly rotating shaft aligned centrally down the tube.

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5. A process as claimed in Claim 4 wherein the blades, arms or sweeps are so adjusted as to pass through the film of liquid.

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6. A process as claimed in any one of Claims 2 to 5 wherein the cylindrical tube is open ended at the end away from the inlets.

7. A process as claimed in any of the preceding claims wherein the first stage is carried out at a pressure higher than atmospheric.

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8. A process as claimed in any of the preceding claims wherein a polyamide is used and the product is an organic polyisocyanate.

9. A process as claimed in any of the preceding claims wherein the solution of amine or polyamine in inert solvent contains between 4% and 16% by weight of amine or polyamine.

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10. A process according to Claim 1 as hereinbefore described with reference to any one of the Examples.

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11. Organic isocyanates whenever manufactured by a process as claimed in any one of the preceding claims.

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BERTRAM F. DREW,  
Agent for the Applicants.

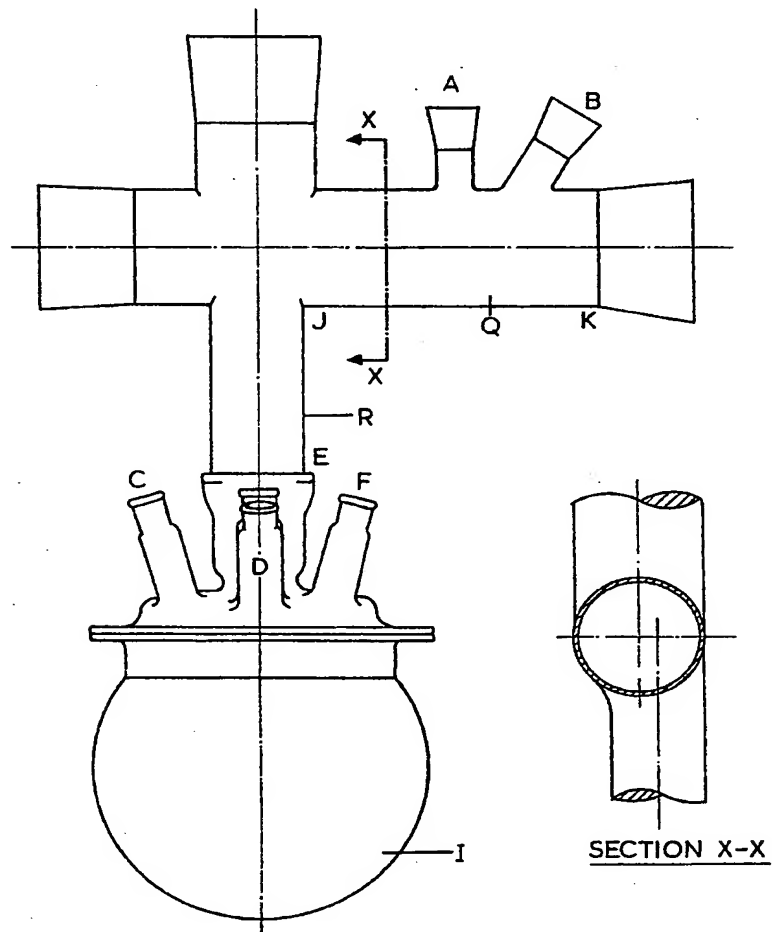
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PROVISIONAL SPECIFICATION

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Sheet 1*

**FIG. 1**  
**COLD STAGE REACTOR WITH HOT STAGE**  
**REACTOR ATTACHED**



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PROVISIONAL SPECIFICATION

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sheet 2*

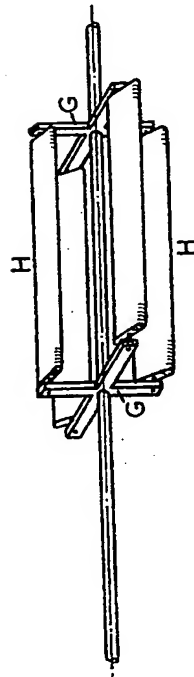
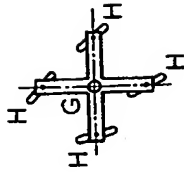


FIG. 2.

COLD STAGE REACTOR AGITATOR



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PROVISIONAL SPECIFICATION

3 SHEETS

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Sheet 3

